Enhanced oxidation resistance of Ti-rich Mo-Si-B alloys by pack-cementation process

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Enhanced oxidation resistance of Ti-rich Mo-Si-B alloys by pack-cementation process

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Motivation

- **Mo-Si-B alloys**
  - Possess oxidation resistance and good creep resistance at 1200 °C
  - Suffer from relatively high density (9.6 g/cm³)

- Potential alternatives are Ti-rich Mo-Si-B alloys
  - Reduced density (7.8 g/cm³)
  - Higher creep resistance
  - Lower oxidation resistance (formation of mixed SiO₂·TiO₂ duplex oxide scale)

- Improve oxidation behavior of Ti-rich Mo-Si-B alloys

- Pack-cementation previously successfully applied to Mo-Si-B based alloys to enhance oxidation resistance

- Transfer the process to Mo-Si-B-Ti
Material and experimental procedure

- Substrate to be coated: Mo-12.5Si-8.5B-27.5Ti (in at.%)  
  - Prepared by arc-melting in Ar-atmosphere  
  - Heat-treated at 1600 °C for 100 h

- Pack-cementation process / two-step process

  1) Pack-coating at 1000 °C for 40 h in Ar-atmosphere
  2) Conditioning at 1400 °C for 10 h in air

Powder mixture:
35 wt.% Si + B
2.5 wt.% NaF
62.5 wt.% Al₂O₃
Material and experimental procedure

- Substrate to be coated: Mo-12.5Si-8.5B-27.5Ti (in at.%)
  - Prepared by arc-melting in Ar-atmosphere
  - Heat-treated at 1600 °C for 100 h

- Pack-cementation process / two-step process
  1) Pack-coating at 1000 °C for 40 h in Ar-atmosphere
  2) Conditioning at 1400 °C for 10 h in air

- Investigation
  - Cyclic oxidation experiments: between T (800, 1100, 1200 °C) and 25 °C
    → Oxidation kinetics up to 1000 h
  - Microstructure examination
    → Characterize layer development
Microstructure: layer after step 1

Si+B pack-cementation on Mo-12.5Si-8.5B-27.5Ti

- Layer consists of MoSi₂ with a Ti content of 13 at.%
- No equilibrium between MoSi₂ layer and substrate

Si+B pack-cementation on Ti-free Mo-alloy

- Outer layer composed of MoSi₂ while inner layer is MoB
- MoB layer after cementation favors formation of Mo₅SiB₂ – acts as a diffusion barrier

Microstructure

Layer after conditioning (step 2)
Mo-12.5Si-8.5B-27.5Ti

Three separate layers
→ Oxide scale and two diffusion layers
**Microstructure**

**Layer after conditioning (step 2)**
Mo-12.5Si-8.5B-27.5Ti

- Three separate layers
- Oxide scale and two diffusion layers

**Layer after cyclic oxidation test**
1100 °C for 500 h

- MoSi$_2$ layer still present
- No spalling
- Coating intact after thermal cycling
Oxidation behavior: Effect of temperature

Weight change

specific weight change in mg/cm²

-4 -2 0 2

800 °C
1100 °C
1200 °C

0 200 400 1000

time in h
Oxidation behavior: Effect of temperature

Weight change
Self-healing

specific weight change in mg/cm² versus time in h

800 °C
1100 °C
1200 °C
Oxidation behavior: Effect of temperature

Weight change
Self-healing
Temperature effect

specific weight change in mg/cm²

800 °C
1100 °C
1200 °C

0
2

-2
-4

0 200 400 1000

time in h

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Oxidation behavior: Summary

The diagram illustrates the specific weight change in mg/cm² over time in hours for different temperatures: 800 °C (black squares), 1100 °C (blue circles), and 1200 °C (red triangles). The T_oxidation is indicated with green arrows.

Key observations:
- Less initial weight loss
- Earlier transition to mass gain

The graph shows that the specific weight change decreases with time for all temperatures, indicating oxidation. At higher temperatures, the weight loss occurs more rapidly, leading to earlier transition to mass gain.
Oxidation behavior: benefit of coating

Significant improvement of oxidation resistance

Summary

- Same Pack-cementation process used for ternary Mo-Si-B can be also applied to Ti containing alloy, but it has a benefit of density.

- Increasing temperatures
  - initial mass loss decrease
  - earlier turnover to mass gain

- Formation of a three layer coating during conditioning
  - consisting of borosilicate, MoSi$_2$ and Ti$_5$Si$_3$

- Layers are intact after thermal cycling and no spalling
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